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Chlorine adsorption kinetics of activated carbon from selected local raw materials

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Chlorine adsorption kinetics of activated carbons from Cattle bone, coconut-shell and wood as influenced by mode of activation (heat and acid) were studied using a commercial activated carbon (CAC) as control. Batch chlorine adsorption by each activated carbon was investigated by contacting with fixed amount of chlorine solution at 10, 15, 20, 25, and 30°C respectively at 10 min intervals. The residual chlorine data were fitted with Freundlich, Langmuir isotherms, and first order kinetic model. The data were also fitted to Fick's unsteady state mass transfer equation and chlorine diffusivities were calculated from the slopes. Heat (H) activation was at 950°C for 3 h to produce heat activated bone carbon (HCB), and 850°C for 2.5 h for heat activated coconut-shell and wood carbon (HCS and HWC) while concentrated phosphoric acid (A) (1:3-1:4) carbon: acid (w/w) was at 600°C for 2 h to produce acid activated wood (AWC), acid activated coconut-shell (ACS) and acid activated bone (ACB) samples. The rates of chlorine adsorption were faster within the first 10-30 min; follow first order kinetics and Freundlich models. Chlorine diffusivities ranged from $3.674 \times 10^{-6} \text{ m}^2/\text{s}$ for Acid activated cattle bone carbon to $5.017 \times 10^{-6} \text{ m}^2/\text{s}$ for Commercial activated carbon. Porosity characterization of the carbons shows a wide range of pore sizes for both gas and liquid phase applications. This study indicated that efficient activated carbons could be obtained from local raw materials by controlled activation with acid or heat for home and industrial purposes.

Key words: Adsorption, kinetics, activations, isotherms, diffusivities, porosity.

INTRODUCTION

The importance and relevance of activated carbon to an ever growing society cannot be overemphasized considering its enormous uses. Its uses range from liquid phase to gaseous phase applications. One of the important applications of activated carbon is in water dechlorination. Chlorination is the process of adding the element chlorine to water as a method of water

purification. Chlorine is widely popular for this application because of its ability to kill bacteria and other disease-causing organisms at relatively low concentrations. The chlorine binds and destroys the outer surface of bacteria and viruses thereby preventing waterborne diseases. The chlorine that is not used is called free residual chlorine. There are many health concerns regarding residual chlorine in water. This is mainly because chlorine is not naturally needed by the body and as a halogen, it tends to inactivate enzymes (Strands, 2008). Chlorinated water contains chemical compounds called trihalomethanes which are carcinogens. Bathing with chlorinated water could have the same effect as drinking because of dermal absorption of chlorine (Nedalee and Abdullah, 2009). Other concerns are aesthetic such as chlorine taste and odour. These concerns inform the need for an

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Abbreviations: **ACB**, Acid activated bone carbon; **ACS**, Acid activated coconut shell carbon; **AWC**, Acid activated wood carbon; **CAC**, Commercial activated carbon; **HCB**, Heat activated bone carbon; **HCS**, Heat activated coconut shell carbon; **HWC**, Heat activated wood carbon.

effective means of water de chlorination after disinfection at low cost. In Europe, the most common means of destroying microorganisms in water system is the use of ozone treatment. In Nigeria, chlorine is extensively used in water treatment because it is cheaper than ozone. Activated carbons are good at removing chlorine residual from water but the actual rates of chlorine removal by activated carbons obtained from different local raw materials need to be determined. Fast adsorption rate and high adsorption capacity are important and must be considered in the selection of an activated carbon for a given purpose. The performance of activated carbon is indicated by its adsorptive characteristics, which is derived from the specific surface area, pore size and pore volume of the carbon (Akinyemi and Taiwo, 2004). In this study, adsorption isotherms and chlorine removal kinetics from water using activated carbon from selected local raw materials were considered.

MATERIALS AND METHODS

Source/sample preparation

Coconut shells, wastes pieces of hard wood *Prosopis Africana* were respectively collected from coconut and fire wood sellers in Idah, Kogi state while cattle bones were collected from an abattoir opposite Federal Polytechnic, Idah, kogi state -Nigeria. Each material was crushed manually to small chips of approximately equal sizes (1.0 - 0.8 cm). Analytical grade- Calcium hypochlorite, free chlorine reagent, and a commercial activated carbon (Chemivivon, Sussex, UK) were purchased from Charlitech Laboratories, Makurdi, Nigeria.

Carbonization and activation

Carbonization was carried out using the method described by Gimba et al. (2001). The carbonization time was varied between 0.5 and 3 h at 600°C to determine optimum carbonization for each raw material by fixed carbon measurement without ash formation. Acid activation was carried out using the method described by Xu and Liu (2008). However, the carbon: acid ratio was varied between 1:1 and 1:6 to determine the optimum activation for each of the raw material by iodine value measurement. Heat activation was carried out using the method described by Ajayi and Olawale (2009) and the activation temperature was varied between 700 – 950°C to determine optimum activation by iodine value measurement.

Chlorine adsorption experiment and order of adsorption

The experimental procedure basically involves batch adsorption performed by shaking specified carbon sample in 100 ml of chlorine solution in a glass beaker using an SMZ Stuart magnetic stirrer (Stuart Scientific Co Ltd; Britain) with an intensity of agitation of 6 rps. The experiments were performed by adding a specified amount of carbon to each of the 5 beakers, the beakers were labelled 1-5. Beaker one was placed in a thermostat set to 30°C, while beakers 2, 3, 4 and 5 were subjected to 25, 20, 15 and 10°C respectively for temperature variation. Under all the varied experimental conditions, concentrations of free chlorine remaining in solution after shaking and decanted at the specified time were determined photometric ally using multipara meter bench photometer (manufacturer-Hanna Co. Ltd, Hungary).

The chlorine adsorption was modelled to 1st order adsorption using the equation:

$$dC/dt=KC; \quad (1)$$

Integrating equation (1) gives:

$$\ln C=\ln C_0-Kt \quad (2)$$

Where C is chlorine concentration at time t, C₀ is the concentration at time zero and K is the rate constant.

Adsorption isotherm and determination of chlorine diffusivity (rate of diffusion)

The chlorine adsorption data at the temperature of (30°C) were fitted to two most familiar isotherm equations which characterizes most adsorption processes rather well under conditions present in most water treatment systems (Ademoroti, 2003). The Langmuir and Freundlich constants were calculated from the slopes and intercepts of the isotherms respectively (Okuo et al., 2008). Chlorine diffusivities were calculated from the slopes of Fick's unsteady state mass transfer plots.

The Langmuir equation can be written as:

$$C/N=C/K_2+1/K_1K_2 \quad (3)$$

C is the concentration; K₁ and K₂ are Langmuir constants
The Freundlich equation takes the form:

$$N=KC^n \quad (4)$$

The linear form of the equation can be written as:

$$\log N=\log K+n\log C \quad (5)$$

Where N is the amount of chlorine adsorbed by a unit mass of activated carbon, C is the concentration, K and n are Freundlich constants.

Fick's unsteady state mass transfer plot of $\ln C - C_e / C_0 - C_e$ against time (t)

Where C = instantaneous chlorine concentration, C₀ = the initial chlorine concentration, C_e = the equilibrium chlorine concentration.
The slope K is given as:

$$K=D\pi^2/4L^2 \quad (6)$$

D is the diffusivity (m²s⁻¹) and L is the thickness of the material (m)

Porosity characterization

This was performed by the adsorption of N₂ at 77 k using micrometric surface area analyser (ASAP2010) Micrometric Inc. USA. Before the adsorption of N₂, the samples were subjected to degassing for 2 h at final pressure of 133.32 x10⁻⁴ pa. The total pore volume (V_T) was estimated from the volume of N₂ (as liquid) adsorbed at relative pressure of (p/p⁰) of 0.95 (Xu and Liu, 2008). The mesopore volumes were calculated from adsorption isotherms and mesopore was calculated as the difference between total pore volume and micropore volume.

Statistical analysis

Experimental results were analysed statistically. Means were

Table 1. Regression parameters for first-order chlorine adsorption.

Carbon material	Treatment		Temperature (°C)				
			30	25	20	15	10
Wood	Heat	N	5	5	5	5	5
		r^2	0.999	0.989	0.999	0.999	0.999
		A	1.078	1.081	1.096	1.128	1.137
	Acid	N	5	5	5	5	5
		r^2	0.999	0.999	0.999	0.998	0.994
		A	1.086	1.075	1.072	1.097	1.085
Coconut-shell	Heat	K	0.056	0.058	0.062	0.068	0.072
		N	5	5	5	5	5
		r^2	0.999	0.998	0.999	0.998	0.999
	Acid	A	1.086	1.075	1.072	1.097	1.085
		K	0.047	0.048	0.052	0.055	0.058
		N	5	5	5	5	5
Bone	Heat	r^2	0.997	0.998	0.999	0.999	0.999
		A	1.135	1.000	1.126	1.136	1.137
		K	0.042	0.044	0.048	0.050	0.051
	Acid	N	5	5	5	5	5
		r^2	0.999	0.998	0.999	0.998	0.999
		A	1.116	1.089	1.102	1.110	1.110
Commercial carbon	Heat	K	0.042	0.044	0.048	0.051	0.053
		N	5	5	5	5	5
		r^2	0.999	0.999	0.999	0.999	0.989
	Acid	A	1.135	1.137	1.115	1.123	1.130
		K	0.041	0.042	0.046	0.048	0.050
		N	5	5	5	5	5
Acid	r^2	0.999	0.999	0.999	0.998	0.994	
	A	1.126	1.117	1.087	1.095	1.107	
	K	0.041	0.043	0.046	0.048	0.050	
Commercial carbon	N	5	5	5	5	5	
	r^2	0.985	0.999	0.999	0.999	0.989	
	A	1.107	1.124	1.161	1.162	1.052	
		K	0.060	0.065	0.073	0.077	0.082

N = Number of entries, r = Correlation coefficient, A = Intercept, and k = Rate constant per minute.

compared using test of significant difference (Steel and Torrie, 1980). The least square linear regression was employed for calculating the gradients and intercepts of the linearized forms of the kinetic models.

RESULTS

The results of regression parameters for the first order adsorption are shown in Table 1. The r^2 values for 1st order ranged from 0.998 - 0.999. The rate constants (per minute) for heat activated wood carbon ranged from 0.056 - 0.072, coconut-shell carbon 0.042 - 0.051, and bone 0.041 - 0.050. For acid activated carbons, the rate constants ranged from 0.042 to 0.052 (wood), 0.042 to 0.050 (coconut-shell), and 0.047 to 0.058 (bone) as compared to 0.060 to 0.082 for the commercial activated carbon. The regression parameters for Freundlich and

Langmuir adsorption isotherm is shown in Table 2. The r^2 values for Freundlich isotherm ranged from 0.982 for CAC - 0.999 for HWC and ACS while for Langmuir isotherm it ranged from 0.834 for AWC - 0.994 for HWC.

The Freundlich adsorption exponents (n) ranged from 1.218 for AWC to 1.678 for CAC while the adsorption coefficient ranged from 1.062 for HCB to 2.244 for CAC. There was no significant ($P \geq 0.05$) difference between the Freundlich adsorption exponent of CAC and those of HWC, HCS, ACB and HCB samples. On the other hand, there is no Significant ($P \geq 0.05$) difference between the Freundlich adsorption coefficient (K) of CAC and that of HWC. The equilibrium parameter (R_L) indicating adsorptions were 0.394, 0.099, 0.335, 0.214, 0.249, 0.241 and 0.063 for AWC, HWC, ACS, HCS, ACB, HCB and CAC respectively. There was no significant ($P \geq 0.05$) difference between R_L values of CAC and those of HWC

Table 2. Regression parameters for Freundlich and Langmuir isotherm.

Model	Regression parameter	Activated carbon sample							
		AWC	HWC	ACS	HCS	ACB	HCB	CAC	LSD (0.05)
Freundlich	N	4	4	4	4	4	4	4	-
	r^2	0.995	0.999	0.999	0.983	0.988	0.996	0.985	-
	n	1.218	1.527	1.252	1.538	1.486	1.522	1.678	0.221
	K	1.875	2.178	1.493	1.138	1.112	1.062	2.244	0.339
Langmuir	N	4	4	4	4	4	4	4	-
	r^2	0.834	0.994	0.991	0.916	0.921	0.932	0.983	-
	K_1	3.637	2.012	3.493	1.949	2.114	1.992	1.751	-
	K_2	0.804	3.049	0.662	1.221	1.004	1.050	4.966	-
	R_L	0.394	0.099	0.335	0.214	0.249	0.241	0.063	0.163

N= Number of entries, r = correlation coefficient, n = Adsorption exponent and K is a measure of adsorption capacity, K_1 = Langmuir adsorption capacity, K_2 is the Langmuir constant related to energy of adsorption and R_L is an indicator of adsorption

and HCS while AWC, ACS, ACB and HCB had statistically similar R_L values. The R_L values ranged from 0.099 for CAC - 0.394 for HAW. The regression parameters for Fick's expression of chlorine adsorption are presented in Table 3. Variation of chlorine diffusivities with temperatures is presented in Figure 1. Chlorine diffusivities increase with decreased in temperature for all the carbons. Diffusivities (m^2/s) decreased from 3.559×10^{-6} - 3.968×10^{-6} for AWC, 3.909×10^{-6} - 4.667×10^{-6} , for HWC, 3.209×10^{-6} - 3.784×10^{-6} for ACS, 3.092×10^{-6} - 3.617×10^{-6} for HCS, 3.209×10^{-6} - 3.675×10^{-6} for ACB, 3.267×10^{-6} - 3.675×10^{-6} for HCB and 4.084×10^{-6} - 5.017×10^{-6} for CAC respectively. The pore size distribution curves of the carbons are presented in Figure 2.

DISCUSSION

The high correlation coefficients in addition to linearity of the curves (Table1) indicated that chlorine removal from water by the activated carbons can be modelled using first order kinetics. Earlier studies indicated that first order kinetic model adequately described chlorine desorption by activated woody pod carbon and silica (Ejikeme and Ochonogor, 2008), (Sakamota et al., 1994). The results presented in Table 2 shows Langmuir isotherm is essential in assessing the adsorption efficiency of adsorbent. This model was based on assumption of formation of its initial chlorine monolayer of adsorb ate species onto the surface of adsorbent. This study is useful in optimizing the operating condition for effective adsorption. Equilibrium parameter R_L which is considered as a more reliable indicator of adsorption (Omonmhenle et al., 2008), (McKay and McConvey, 1985) was computed from the expression represented as follows:

$$R_L = \frac{1}{1+K_2} \quad (7)$$

Where C initial chlorine concentration (mg/l), K_2 is the Langmuir constant values between 0&1 indicate favourable adsorption for all initial concentration and temperature studied. R_L indicates isotherm shape and whether it is favourable or not as per the criteria given subsequently (Inamullah et al., 2008).

Adsorption isotherm is the relationship between the amount of substance adsorbed by an adsorbent and the equilibrium pressure or concentration at constant temperature. The isotherms show that correlation coefficients (r) for Freundlich and Langmuir were high. The high correlations suggest that both isotherms describe chlorine sorption on the activated carbons with comparable success, with the Freundlich model being superior. An indicator of adsorption R_L from Langmuir constants indicates favourable adsorption for all carbons at temperatures studied (Table 4). The lower the R_L values, the more favourable the adsorption process. R_L indicates isotherm shape and whether it is favourable or not as per the criteria given subsequently (Geetha et al., 2008). In a related study, favourable adsorption of 2-4-Dichlorophenol on soil activated samples with R_L values between 0.649 to 0.719 for different treated clay samples were reported (Omonmhenle et al., 2008). Several researchers have reported the applicability of Freundlich and Langmuir isotherms in carbon (Okuo et al., 2008), (Onogwu et al., 2001), (Ejimeke and Ochonogo, 2008), (Ufuah et al., 2004). The K and n from Freundlich isotherm and the K_1 and K_2 from Langmuir isotherm incorporates all the factors affecting adsorption capacity and intensity. It has been suggested that n values of between 1 and 10 represent favourable adsorption (Ufuah et al., 2004).

The increase in diffusivities with decrease in temperature (Figure 1) could be attributed to exothermicity of adsorption (Okuo and Ozioko, 2001). Adsorption is an exothermic process and is favoured by lower energy. In addition, at lower temperature, more of the chlorine exists in adsorb able

Table 3. Regression paramers for ficks unsteady state mass transfer.

Carbon sample	Treatment	Parameter	Temperature(°C)					
			30	25	20	15	10	
Wood	Heat	Entries	6	6	6	6	6	
		SD Error	0.016	0.016	0.015	0.015	0.014	
		Slope	0.067	0.070	0.072	0.072	0.080	
			Diffusivity(m ² /s)	3.909 × 10 ⁻⁶	4.111 × 10 ⁻⁶	4.200 × 10 ⁻⁶	4.434 × 10 ⁻⁶	4.667 × 10 ⁻⁶
	Acid	Entries	6	6	6	6	6	
		SD Error	0.015	0.015	0.016	0.014	0.014	
		Slope	0.061	0.062	0.065	0.065	0.066	
Diffusivity(m ² /s)		3.559 × 10 ⁻⁶	3.617 × 10 ⁻⁶	3.792 × 10 ⁻⁶	3.852 × 10 ⁻⁶	3.968 × 10 ⁻⁶		
Coconut-shell	Heat	Entries	6	6	6	6	6	
		SD Error	0.015	0.016	0.016	0.015	0.015	
		Slope	0.053	0.056	0.060	0.061	0.063	
		Diffusivity(m ² /s)	3.092 × 10 ⁻⁶	3.267 × 10 ⁻⁶	3.500 × 10 ⁻⁶	3.559 × 10 ⁻⁶	3.617 × 10 ⁻⁶	
	Acid	Entries	6	6	6	6	6	
		SD Error	0.015	0.015	0.016	0.016	0.017	
		Slope	0.055	0.058	0.060	0.062	0.064	
		Diffusivity(m ² /s)	3.209 × 10 ⁻⁶	3.384 × 10 ⁻⁶	3.500 × 10 ⁻⁶	3.639 × 10 ⁻⁶	3.734 × 10 ⁻⁶	
Bone	Heat	Entries	6	6	6	6	6	
		SD Error	0.017	0.016	0.016	0.015	0.015	
		Slope	0.056	0.057	0.060	0.062	0.063	
		Diffusivity(m ² /s)	3.267 × 10 ⁻⁶	3.326 × 10 ⁻⁶	3.403 × 10 ⁻⁶	3.617 × 10 ⁻⁶	3.675 × 10 ⁻⁶	
	Acid	Entries	6	6	6	6	6	
		SD Error	0.015	0.016	0.016	0.016	0.015	
		Slope	0.055	0.056	0.060	0.062	0.063	
		Diffusivity (m ² /s)	3.209 × 10 ⁻⁶	3.267 × 10 ⁻⁶	3.403 × 10 ⁻⁶	3.617 × 10 ⁻⁶	3.675 × 10 ⁻⁶	
Commercial carbon		Entries	6	6	6	6	6	
		SD Error	0.014	0.014	0.013	0.014	0.014	
		Slope	0.070	0.073	0.080	0.081	0.086	
		Diffusivity(m ² /s)	4.084 × 10 ⁻⁶	4.259 × 10 ⁻⁶	4.667 × 10 ⁻⁶	4.726 × 10 ⁻⁶	5.017 × 10 ⁻⁶	

form and more adsorption rather than desorption occurs. The diffusivities (m²/s) ranged from 5.017 × 10⁻⁶ to 4.667 × 10⁻⁶. The variation in diffusivities of chlorine for the different carbons could be attributed to the differences in structural properties of the carbons (Carolina et al., 1995).

The pore size distribution curves (Figure 2); showed a broader range of pore sizes for ease of movement of adsorbate (chlorine) through the carbon pores and for the adsorption. The BET surface area of the carbons ranged from 1015 for HWC to 782m²g⁻¹ for ACB while that of control (CAC) was 1022 m²g⁻¹. The surface area for the activated carbons fall within the range of 600 to 1200 m²g⁻¹ reported for most widely used commercial activated carbons (Ng et al., 2002). The high surface

area of the carbons is important since it limits the amount of adsorbate which can be adsorbed.

The BET surface areas were higher for acid activated carbons and this is expected because chemical activation normally develops more porosity and gives high surface area when compared with thermal activation (Ajayi and Olawale, 2009). The increase BET surface areas and microspore (0.746 to 0.641cm³g⁻¹) structure of acid activated carbons was brought about by oxidation of the carbons by phosphoric acid. The macrospore volume of the carbons ranged from 0.779 for HWC- 0.656cm³g⁻¹ for HCB and the mesopore volume ranged from 0.008 for HWC – 0.040 cm³g⁻¹ for HCS. The broader pore size for these carbons is important for a wide application since pore volume limits the size of molecules to be adsorbed.

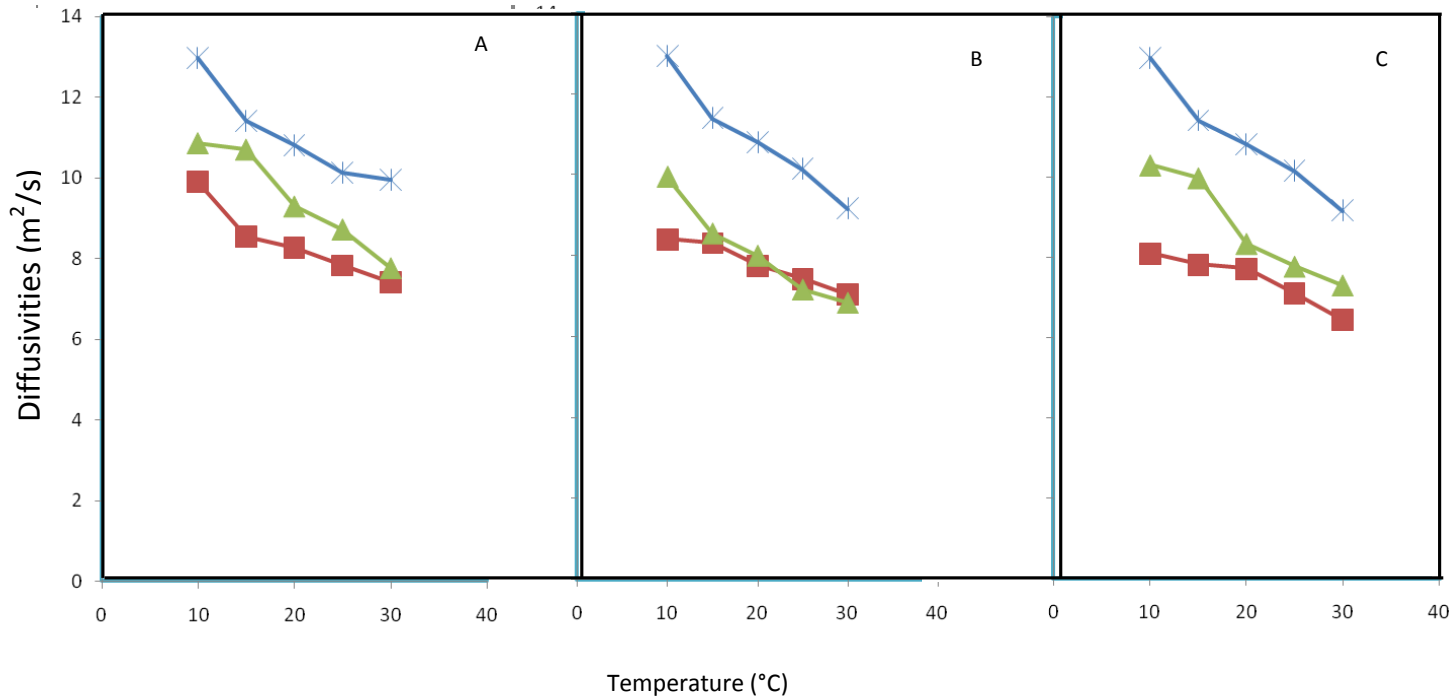


Figure 1. Effect of temperature on chlorine diffusivities by commercial carbon (*), heat activated (▲) and acid activated (■) carbons from wood (plot A), coconut-shell (plot B) and bone (plot C) respectively.

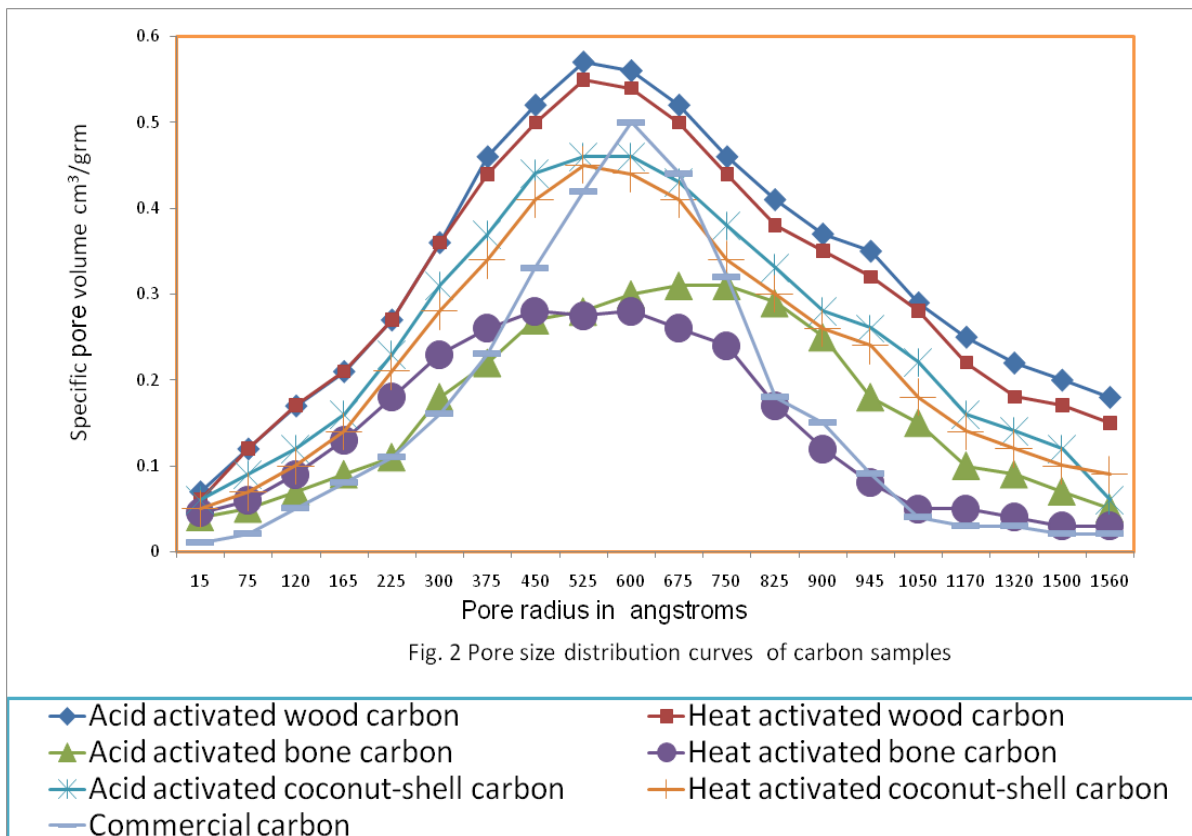


Fig. 2 Pore size distribution curves of carbon samples

Figure 2. Pore size distribution curves of carbon samples.

Table 4. Adsorption isotherm.

RL values	Adsorption
RL > 1	Unfavourable
RL = 1	Linear
0 < RL < 1	Favourable
RL = 0	Irreversible

Conclusion

This study indicates that efficient activated carbons can be obtained from bone, wood and coconut shell by controlled activation with phosphoric acid or heat for water de chlorination and for other uses. The adsorption of chlorine by the carbons compared favourably with a commercial carbon and from chlorine diffusivity (D) values, phosphoric acid activation is the best for coconut shell while thermal (heat) activation gives the best results for wood and bone carbon.

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